The Performance of an Ex-Situ Reactor System for TEM Studies of Catalyst Reactions

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An "ex-situ" reactor has been designed for use in exposing transmission electron microscopy (TEM) catalyst specimens to gas mixtures at temperatures that simulate actual use. The optimization of currently available catalysts and the development of new ones requires a detailed understanding of the effects of both microstructure and composition on their function. Previous work has demonstrated that information at the atomic-scale on heterogeneous catalysts can be derived using high resolution transmission electron microscopy. Under favorable conditions, the morphology of heavy metal catalytic particles can be related to catalytic activity. It has been shown that preoxidation can disrupt the surface of small metal particles causing altered activity and selectivity in reactions such as alkane hydrogenolysis. Metals such as Pt, Rh, or Ru are noble and pick up no more than a monolayer of oxygen when exposed to air during sample preparation for microscopy. This oxygen monolayer is not imaged in the microscope, most likely because the oxygen desorbs during exposure to the high energy electron beam. However, when metals such as Fe, Co, Ni, Pd or Cu are exposed to air, there is a corrosive interaction that alters particle structure quite dramatically. To examine the microstructure of such catalysts in their reduced, catalytically active state, it is important to be able to transfer the sample from the reaction environment into the microscope without exposing it to the ambient. The TEM specimen holder/*ex-situ* reactor system permits sensitive catalyst specimens to be mounted on a specimen grid, installed in the reactor, reacted with any chosen gas at atmospheric pressure and temperatures up to 1200°C, then transferred into the Hitachi HF-2000 field emission gun TEM without exposure to the atmosphere. The reactor system design will permit direct imaging of the